

U.S.S.N.: 10/381,690

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Andrew Goodwin et al.
Serial No.: 10/381,690
Group Art Unit: 1762
Filed: March 25, 2003
Examiner: David P. Turocy
For: METHOD AND APPARATUS FOR FORMING A COATING

DECLARATION UNDER 37 CFR § 1.132

Mail Stop AMENDMENT
Commissioner of Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Dear Sir:

I, Dr. Liam O'Neill, hereby state that:

1. I am a citizen of the Republic of Ireland.
2. I have a doctorate from University College Cork in Ireland. I am currently employed as a Research Scientist for Dow Corning Ireland Limited of Cork, Ireland. I have worked in the plasma discharge field for approximately 5 years and I have been employed by Dow Corning Ireland Limited since 2002.
3. I am not an inventor of the pending U.S. patent application, Application No. 10/381,690. However, I am a person skilled in the art of plasmas including, in particular, atmospheric pressure plasma glow discharge (APGD) and I am closely involved in development of plasma technologies within Dow Corning Ireland Limited. The instant invention was

invented, in part, by inventors employed by Dow Corning Ireland Limited.

4. The claimed invention of the '690 application is a method for forming a coating on a substrate comprising the steps of introducing a coating-forming material selected from an atomized liquid or a combination of an atomized liquid and an atomized solid into a homogeneous APGD atmosphere or an ionized gas stream resulting therefrom, and exposing the substrate to the atomized coating-forming material. The claimed invention also includes an apparatus including (I) a device for generating a homogeneous APGD, (II) an atomizer for providing an atomized coating-forming material within the plasma glow discharge, and (III) a device for supplying the coating-forming material.

5. Homogeneous APDG, and devices used to produce homogeneous APGD, didn't appear until the work of Okazaki, Kanazawa, et al. in the late 1980's/early 1990's (see "Glow Discharge Plasma at Atmospheric Pressure and its Application", S. Okazaki et al., Proc. Jpn. Symp. Plasma Chem./ Vol. 2 1989, pgs. 95-102). The conditions for obtaining homogeneous APGD were unknown at the time Okazaki et al. was published, and the conditions for obtaining homogeneous APGD were not obvious. It has even been recognized in the art that the conditions for obtaining homogeneous APGD were not obvious prior to Okazaki et al. (see last full paragraph of the second column on page 2950 of Massines et al., "Experimental and theoretical study of a glow discharge at atmospheric pressure controlled by dielectric barrier", Journal of Applied Physics, 1997), and Kanazawa et al. are credited with discovery of the conditions necessary to obtain stable and homogenous APGD (see *Id*). Therefore, prior to the work of Okazaki and Kanazawa, the conditions necessary to achieve homogeneous APGD were simply unknown and any reference that claims to produce

homogeneous APGD prior to the work of Okazaki and Kanazawa is meaningless to those of skill in the art.

6. The idea of using gases with plasma was well-entrenched in the methodologies of the industry and APGD was generally recognized as a dry process until conception of the present invention. Further, it was generally thought that use of anything other than gases with APGD would result in disruption and destabilization of the APGD such that alternatives were not possible. Further, the specific parameters required to achieve homogeneous APGD that were first discovered by Okazaki and Kanazawa et al. contributed to the general mindset that homogeneous APGD was a sensitive process that could be easily disrupted and destabilized should the process be modified. As such, the combined teachings of Kolluri et al. (which does not teach APGD) and Sayers et al. (which teaches APGD but only teaches that gases are introduced into the APGD) do nothing to change the general mindset of those of skill in the art at the time of the instant invention that APGD was limited to use of gases.

7. With regard to claims 30 and 31 in the instant application, the homogeneous APGD may be used to effect controlled free radical induced polymerization of a monomer (which monomer, at least in the context of claims 30 and 31, is present in the coating-forming material). With regard to claims 30 and 31, the homogeneous APGD must be a non-thermal equilibrium plasma (often referred to as a “cold” plasma), which impacts no significant thermal energy to the coating-forming material introduced therein. By impacting no significant thermal energy to the coating-forming material introduced into the plasma, a substantial portion of chemical bonds in the coating forming material remain unbroken. Because the substantial portion of chemical bonds in the monomer remain unbroken,

polymerization of the monomer remains possible after passing through the plasma. Further, by impacting no significant thermal energy to the coating-forming material introduced into the plasma, the physical state of the coating-forming material remains unchanged (i.e., atomized liquid that is introduced into the plasma remains in the liquid state after passing through the plasma).

8. Thermal equilibrium plasmas operate at high temperatures that are drastically higher than operating temperatures of non-thermal equilibrium plasmas (i.e., thermal equilibrium plasmas typically operate at temperatures of at least about 1000 °C versus about ambient temperature for non-thermal equilibrium APGD). Thermal equilibrium plasmas impart significant thermal energy to the material introduced therein, resulting in fragmentation of the molecules in the material and significant breakage of bonds in the material and therefore cannot be used for polymerization of a precursor.

9. Although U.S. Patent No. 5,366,770 to Wang describes a system in which a precursor is introduced into a plasma as an aerosol, the precursor is physically vaporized in the plasma because the plasmas taught by Wang are high temperature plasmas, which are used throughout Wang to vaporize the precursor. (see Abstract, column 4, line 45, column 5, line 6 and in Claim 1, sections (e), (g), (l) and (m), and column 5, lines 6-10). Vaporization of the atomized liquid precursor in the plasmas of Wang et al. converts the system into a standard plasma enhanced CVD reaction with gas phase precursors. {Abstract, column 4, line 45, column 5, line 6 and in Claim 1, sections (e), (g), (l) and (m)}. Further, due to the amount of energy transferred to the precursor from the plasma, polymerization of precursors is not possible through use of the systems taught by Wang, which is relevant at least to claims

30 and 31. Further still, as set forth in paragraph 5 above, those of skill in the art did not know how to make a homogeneous APGD prior to the work of Okazaki and Kanazawa, et al. such that Wang would not have effectively taught those of skill in the art **how** to produce homogeneous APGD. As such, the system taught by Wang does not adequately teach a homogeneous APGD and, as a result, one of skill in the art with knowledge of homogeneous APGD would **not** be taught to use an atomized liquid in homogeneous APGD systems by considering the teachings of Wang. All of these facts relating to Wang are clearly evident to persons of skill in the art of atmospheric pressure glow discharge.

10. As to the references relied upon by Wang for the plasma processes used therein:

- A. J Mort et al "Plasma Deposited Thin Films" (CRC Press Inc., Boca Raton, Fla, 1986) pre-dates the discoveries by Kanazawa et al and does not describe homogeneous APGD.
- B. Methods of Experimental Physics, Volume 9 – Parts A and B, Plasma Physics (Academic Press, NY, 1970/1971) also pre-dates the discoveries by Kanazawa et al. and does not describe homogeneous APGD.
- C. N.H. Burlingame, "Glow Discharge Nitriding of Oxides", PhD thesis (Alfred Uni., Alfred, NY, 1985) also pre-dates the discoveries by Kanazawa et al. and describes the use of high energy plasma which "allows intense, localized heating of a process material". As such, it does not describe a homogeneous APGD, and the systems taught therein could not be used for polymerization of a precursor.
- D. A Model 56 torch from Tafa Inc. is used for the examples provided. This is a standard plasma spray torch for producing high temperature plasmas. Temperatures within this torch exceed 1700K and, therefore, this torch is not suitable for producing homogeneous APGD.
- E. B. Chapman, "Glow Discharge Processes (John Wley & Sons, New York, 1980) reviews the state of the art in plasma technology in 1980. As such, the review summarises DC and RF glow discharges that occur in vacuum systems and does not disclose the existence of homogeneous APGD systems similar to those of Kanazawa et al.

10. Column 4, lines 48 – 52 of Wang describe a suitable frequency domain for generating the plasma taught therein as being in the range of 100 kilohertz up to 5 megahertz, which is too high to produce homogeneous APGD. To support this conclusion, reference is made to pages 55 to 58 of J.R. Roth, Industrial Plasma Engineering, Institute of Physics, London, 2001, which states that the frequency used to generate APGD is in the range of 0.5 to 40 kHz, which is well below the range reported by Wang. Therefore, Wang does not describe the use of APGD.

11. In summary, the equipment and process described in Wang is significantly different to the equipment and process used to produce the homogeneous APGD for the instant invention. Wang describes processes based on high temperature plasmas that result in fragmentation of gas phase molecules and vaporization of atomized liquid introduced therein, with the resultant deposition composed of small molecular fragments, whereas the instant homogeneous APGD does not fragment or vaporize atomized liquid introduced therein, thereby allowing for complex functionality to be retained in the molecules and also allowing polymerization of the same.

12. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information are believed to be true, and further that these statements were made with the knowledge that willful and false statements and the like are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or patent issued thereon.

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Respectfully submitted,

Liam O'Neill

Dated 18th December '07

Dr. Liam O'Neill